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(54) Aromatic high glass temperature sulfone polymer composition

(57) The invention relates to an aromatic high glass transition temperature polymer composition comprising :

- at least one aromatic sulfone polymer (P);
- at least one oxide of a metal;
- at least one carboxylate of a metal, said carboxylate comprising at least 10 carbon atoms.

Preferably, the aromatic sulfone polymer (P) is chosen among the group consisting of polybiphenyldisulfone, polysulfone, polyphenylsulfone, polyethersulfone, copolymers and mixtures thereof.

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Description

[0001] On one hand, the nature of sulfone polymers is such that a certain amount of extractable sulfate is irremediably present aside the polymers. On the other hand, there is a need for aromatic high glass transition temperature polymers, such as polysulfone, polyethersulfone, and polyphenylsulfone , suitable to be used in applications where extractable ionics must be minimized, like in sensitive electronics applications such as the semiconductor industry. It has been already attempted to develop additive packages for the purpose of lowering the level of extracted sulfate.

[0002] Unfortunately, these additives which reduce the level of extractable sulfate are also submitted to extraction. As a result, these additives lead to further extractable issues. These and other drawbacks are remarkably overcome by the aromatic high glass transition temperature polymer composition of the invention.

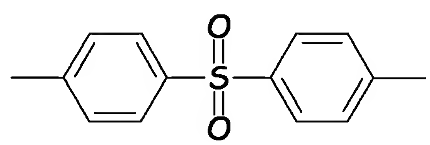
[0003] Thus, it is an object of the invention an aromatic high glass transition temperature sulfone polymer composition comprising :

- at least 90 % by weight, based on the total weight of the composition, of at least one aromatic sulfone polymer (P);
- at least one oxide of a metal;
- at least one carboxylate of a metal, said carboxylate comprising at least 10 carbon atoms.

[0004] Surprisingly, the combination of the oxide and the carboxylate advantageously provides an exceptional efficiency in sequestering the sulfate ion. It is preferred that these additives provide this benefit, and, in addition, they retain the transparent appearance of the aromatic sulfone polymer.

[0005] The composition of the invention comprises preferably at least 92 %, more preferably at least 95 %, still more preferably at least 97.5 % by weight of aromatic sulfone polymer (P), based on the total weight of the composition. For the purpose of the invention, the term "polymer" is intended to denote any material consisting essentially of recurring units, and having a molecular weight above 2000.

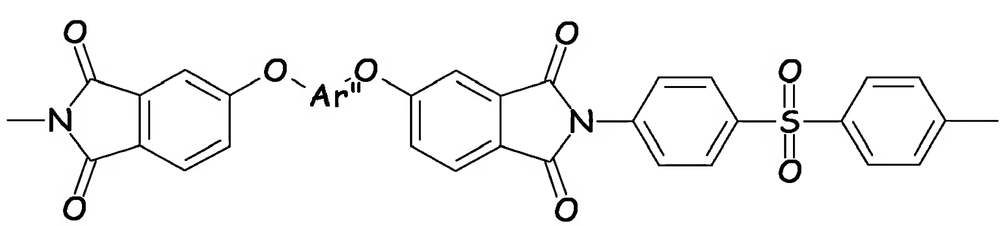
[0006] For the purpose of the invention, the terms "aromatic sulfone polymer (P)" are intended to denote any polymer, at least 50 % wt of the recurring units thereof comprise at least one group of formula 1 :



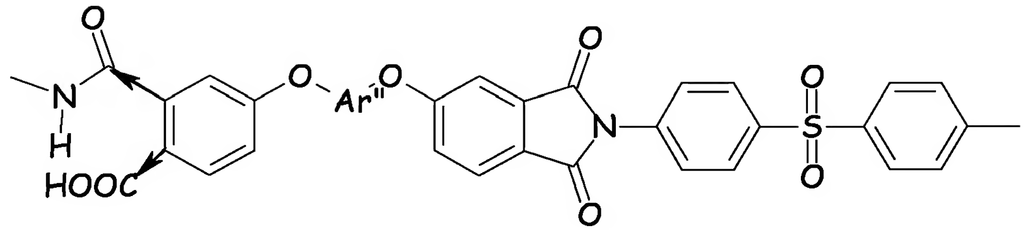
(Formula 1)

[0007] The aromatic sulfone polymer (P) has a glass transition temperature of advantageously at least 150°C, preferably at least 160°C, more preferably at least 175°C.

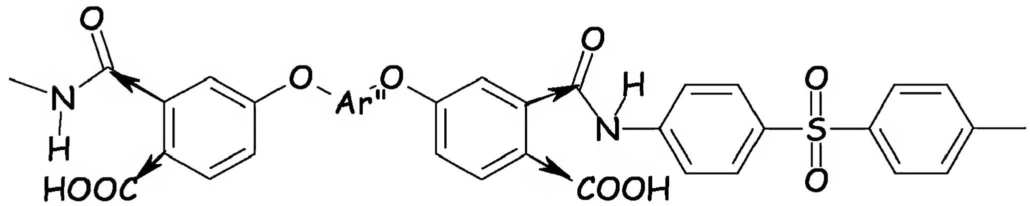
[0008] In a first preferred embodiment of the invention, at least 50 % wt of the recurring units of aromatic sulfone polymer (P) are recurring units (R1), in their imide form (R1-A) and/or amic acid forms [(R1-B) and (R1-C)] :



(R1-A)



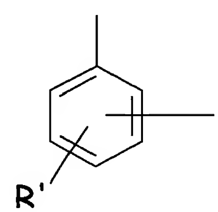
(R1-B)



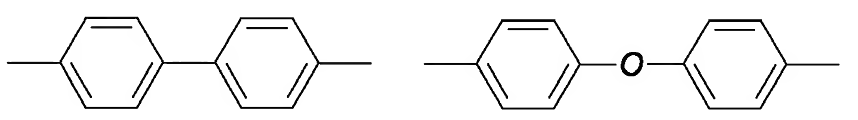
(R1-C)

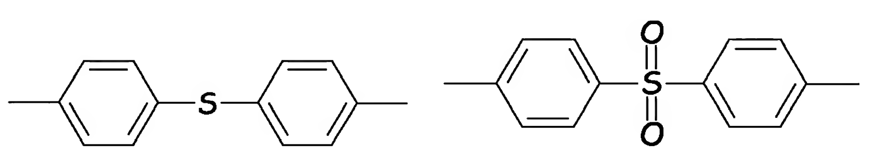
wherein :

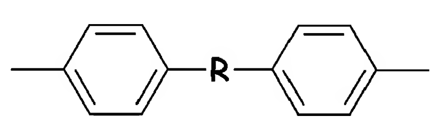
- the → denotes isomerism so that in any recurring unit the groups to which the arrows point may exist as shown or in an interchanged position;
- Ar" is chosen among the following structures :



with the linking groups being in ortho, meta or para position and **R**' being a hydrogen atom or an alkyl radical comprising from 1 to 6 carbon atoms ,

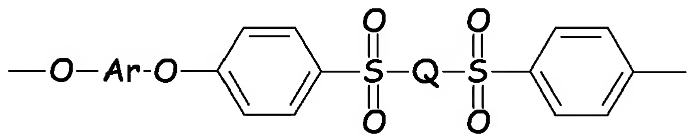




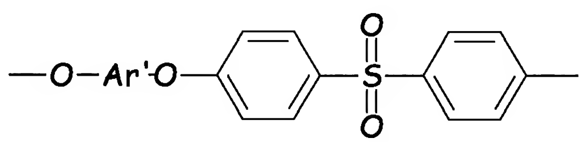


with **R** being an aliphatic divalent group of up to 6 carbon atoms, such as methylene, ethylene, isopropylene and the like, and mixtures thereof.

[0009] In a second preferred embodiment of the invention, at least 50 % wt of the recurring units of aromatic sulfone polymer (P) are recurring units (R2) and/or recurring units (R3) :



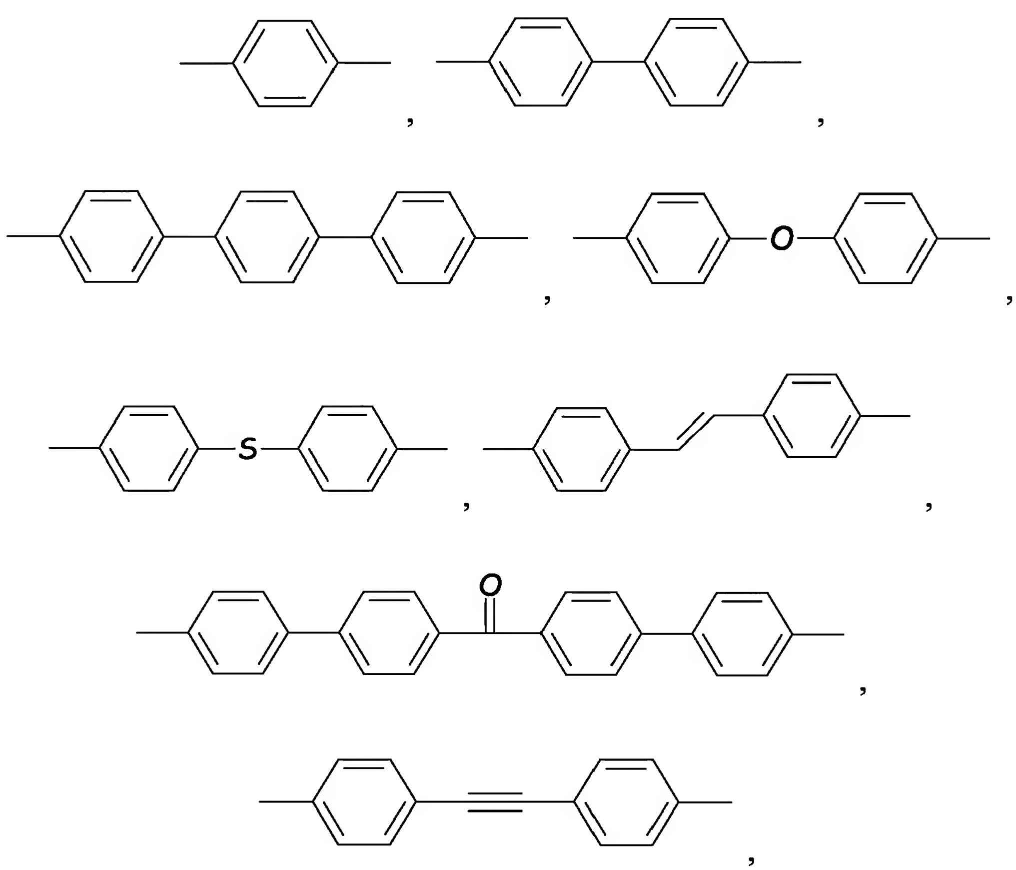
(R2)

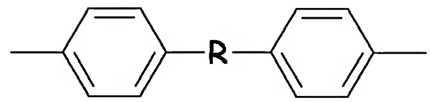


(R3)

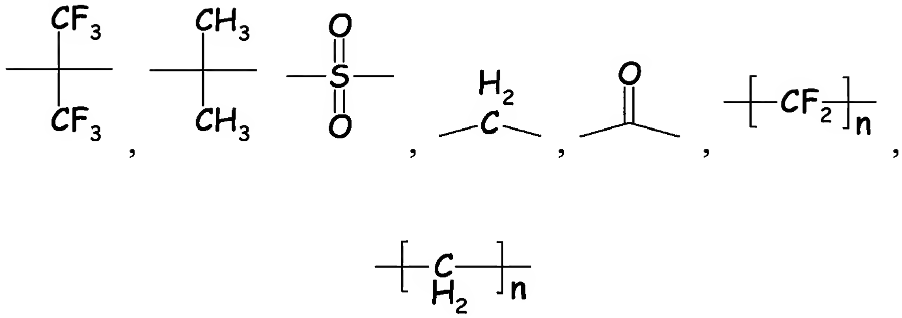
wherein :

- **Q** is a group chosen among the following structures :



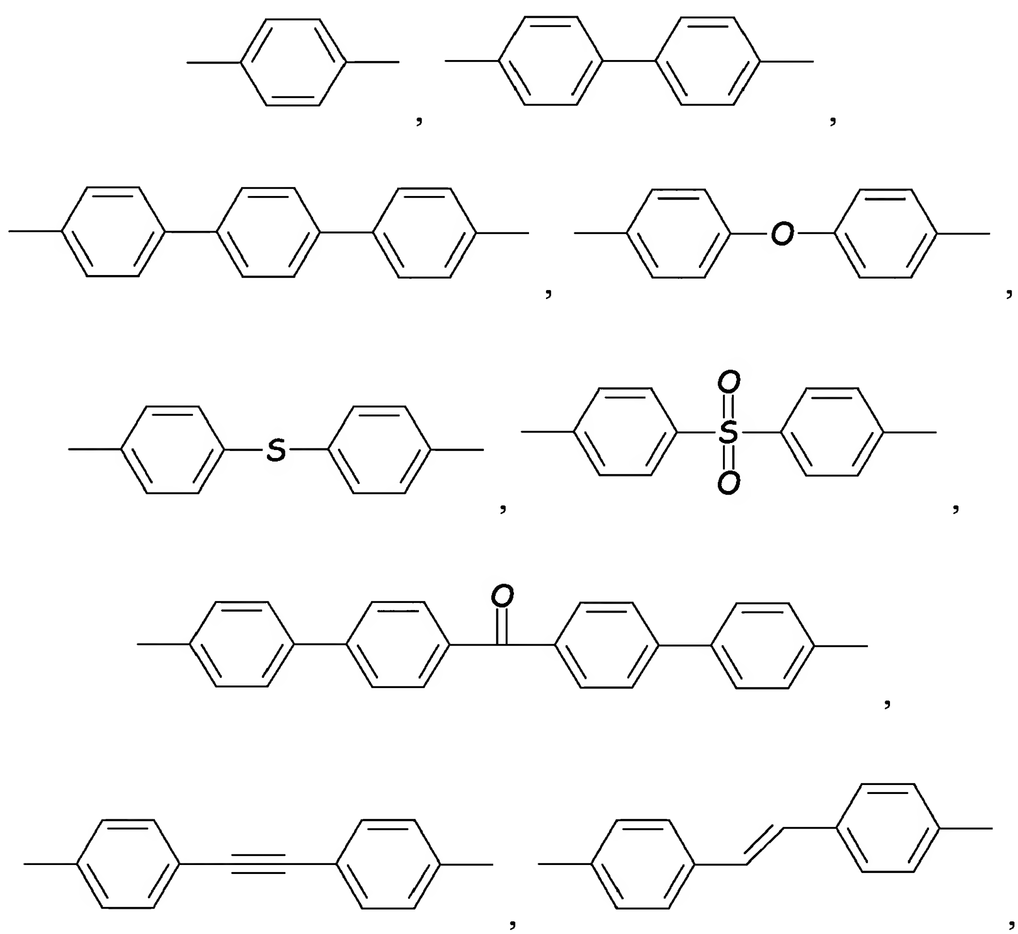


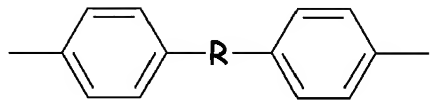
with **R** being :



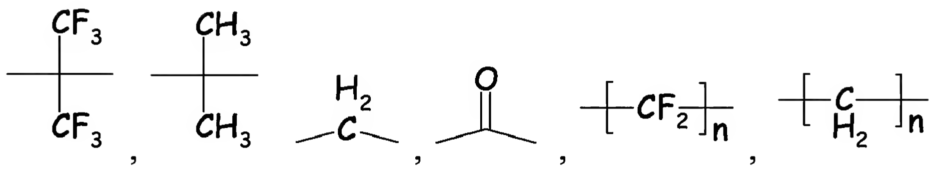
with n = integer from 1 to 6, or an aliphatic divalent group, linear or branched, of up to 6 carbon atoms; and mixtures thereof;

- **A**r is a group chosen among the following structures :



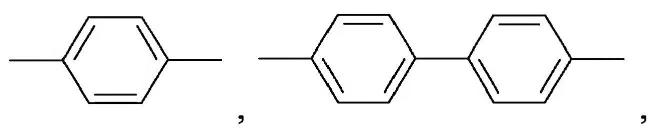


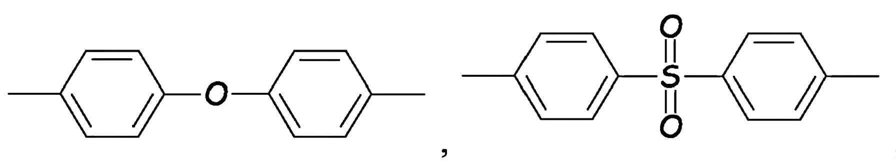
with **R** being :

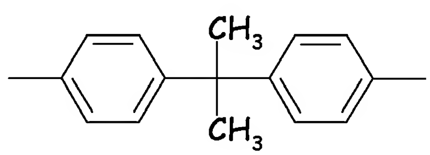


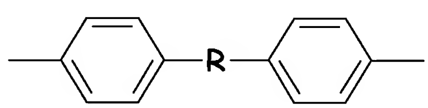
with n = integer from 1 to 6, or an aliphatic divalent group, linear or branched, of up to 6 carbon atoms; and mixtures thereof;

- **Ar**' is a group chosen among the following structures :

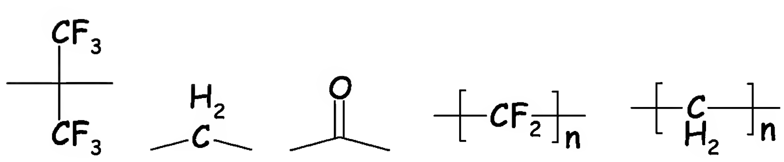






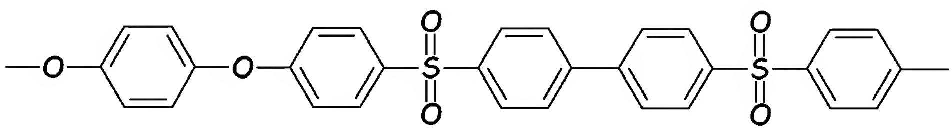


with **R** being :

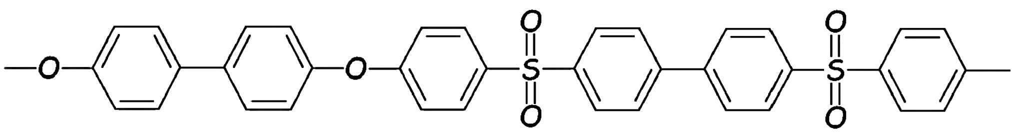


with n = integer from 1 to 6, or an aliphatic divalent group, linear or branched, of up to 6 carbon atoms; and mixtures thereof.

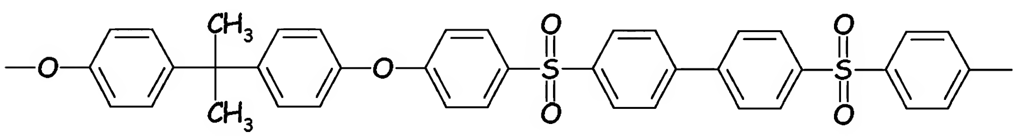
[0010] Recurring units **(R2)** are preferably chosen from :



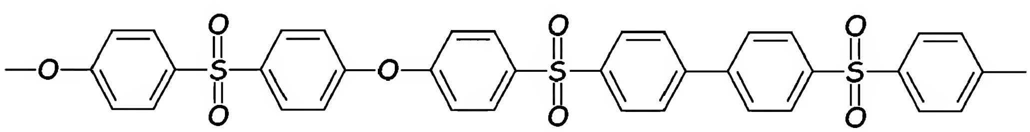
(i)



(ii)



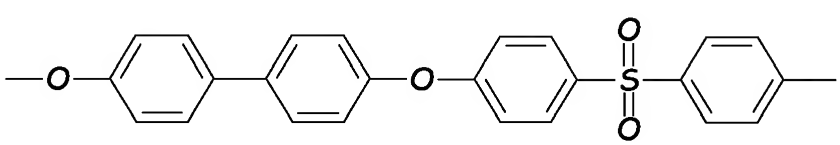
(iii)



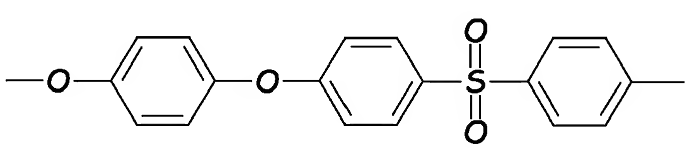
(iv)

and mixtures thereof.

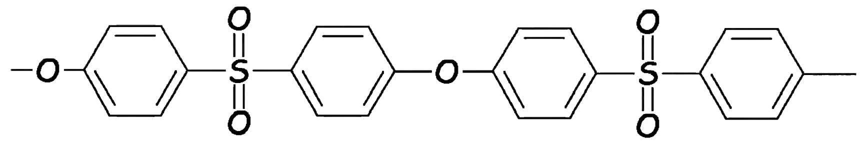
[0011] Recurring units **(R3)** are preferably chosen from :



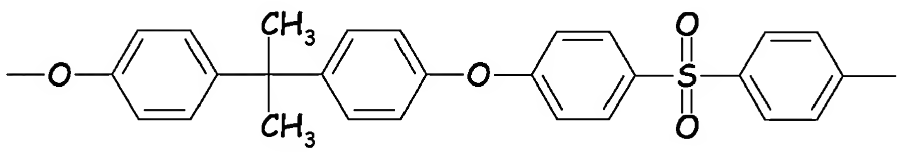
(j)



(jj)



(jjj)



(jv)

and mixtures thereof.

[0012] Aromatic sulfone polymer (P) according to the second preferred embodiment of the invention comprises at least 50 % wt, preferably 70 % wt, more preferably 75 % wt of recurring units (R2) and/or (R3), still more preferably, it contains no recurring unit other than recurring units (R2) and/or (R3).

[0013] Good results were obtained with aromatic sulfone polymer (P) the recurring units of which are recurring units (ii) (polybiphenyldisulfone, herein after), with aromatic sulfone polymer (P) the recurring units of which are recurring units (j) (polyphenylsulfone, hereinafter), with aromatic sulfone polymer (P) the recurring units of which are recurring units (jj) (polyetherethersulfone, hereinafter), with aromatic sulfone polymer (P) the recurring units of which are recurring units (jjj) and, optionally in addition, recurring units (jj) (polyethersulfone, hereinafter), and with aromatic sulfone polymer (P) the recurring units of which are recurring units (jv) (polysulfone, hereinafter).

[0014] Polyphenylsulfone is notably available as RADEL® R PPSF from Solvay Advanced Polymers, L.L.C.. Polysulfone is notably available as UDEL® PSF from Solvay Advanced Polymers, L.L.C.. Polyethersulfone is notably available as RADEL® A PES from Solvay Advanced Polymers, L.L.C..

[0015] Preferably, aromatic sulfone polymer (P) is chosen among the group consisting of polybiphenyldisulfone, polysulfone, polyphenylsulfone, polyethersulfone, copolymers and mixtures thereof.

[0016] Preferably, the oxide of a metal is an oxide of an alkaline-earth metal or of a group 12 transition metal.

[0017] For the purpose of the invention, as group 12 transition metal, we intend designate the group of metals consisting of Zn, Cd, Hg, Uub.

[0018] More preferably, the oxide is chosen among the group consisting of CaO, MgO, ZnO and mixtures thereof. Still more preferably, the oxide is ZnO.

[0019] Preferably the carboxylate of a metal is a carboxylate of an alkaline-earth metal or of a group 12 transition metal.

[0020] More preferably, the carboxylate is chosen among the group consisting of carboxylates of Ca, Mg, Zn and mixtures thereof. Still more preferably, the carboxylate is a Zn carboxylate.

[0021] Preferably, the carboxylate is chosen among caprates (C_{10}), laurates (C_{12}), myristates (C_{14}), palmitates (C_{16}), stearates (C_{18}), arachidates (C_{20}), behenates (C_{22}), palmitoleates (C_{16}), oleates (C_{18}), gadoleates (C_{20}), ricinoleates (C_{18}), linoleates (C_{18}), linolenates (C_{18}), and mixtures thereof. More preferably, the carboxylate is a stearate.

[0022] Excellent results have been obtained with Zn stearate.

[0023] The aromatic high glass transition temperature polymer composition advantageously comprises at least 0.001 % wt, preferably 0.005 % wt, more preferably at least 0.01 % wt of the oxide, based on the total weight of the composition.

[0024] The aromatic high glass transition temperature polymer composition advantageously comprises at most 0.7 % wt, preferably at most 0.6 % wt, more preferably at most 0.5 % wt, even more preferably at most 0.3 % wt, most preferably at most 0.2 % wt of the oxide, based on the total weight of the composition.

[0025] Excellent results were obtained when using from 0.02 to 0.10 % wt, based on the total weight of the composition, of the oxide.

[0026] The aromatic high glass transition temperature polymer composition advantageously comprises at least 0.005 % wt, preferably at least 0.01 % wt, more preferably at least 0.05 % wt of the carboxylate, based on the total weight of the composition.

[0027] The aromatic high glass transition temperature polymer composition advantageously comprises at most 1.00 % wt, preferably at most 0.75 % wt, more preferably at most 0.50 % wt of the carboxylate, based on the total weight of the composition.

[0028] Excellent results were obtained when using from 0.05 to 0.35 % wt, based on the total weight of the composition, of the carboxylate.

[0029] The weight ratio between the oxide and the carboxylate in the composition of the invention is advantageously at most 1 wt/wt, preferably at most 0.75 wt/wt, more preferably at most 0.5 wt/wt.

[0030] The weight ratio between the oxide and the carboxylate in the composition of the invention is advantageously at least 0.05 wt/wt, preferably at least 0.075 wt/wt, more preferably at least 0.10 wt/wt.

[0031] Excellent results were obtained with weight ratios between the oxide and the carboxylate from 0.1 to 0.5 wt/wt.

[0032] Optionally, the high glass transition temperature sulfone polymer composition of the invention can further comprise fillers, lubricating agents, heat stabilizer, anti-static agents, extenders, reinforcing agents, organic and/or inorganic pigments like TiO_2, carbon black, antioxidants, flame retardants, smoke-suppressing agents.

[0033] The composition of the invention advantageously comprises at least one filler chosen from reinforcing fillers, structural fibers and mixtures thereof. Structural fibers may include glass fiber, carbon or graphite fibers, and fibers formed of silicon carbide, alumina, titania, boron and the like, and may include mixtures comprising two or more such fibers. Reinforcing fillers which can also be used in the composition of the invention include notably pigments, flake, spherical and fibrous particulate filler reinforcements and nucleating agents such as talc, mica, titanium dioxide, potassium titanate, silica, kaolin, chalk, alumina, mineral fillers, and the like. The reinforcing fillers and structural fibers can be used alone or in any combination.

[0034] Another aspect of the present invention concerns a process for manufacturing the high glass transition temperature sulfone polymer composition as above described, which comprises mixing :

- at least one aromatic sulfone polymer (P);
- at least one oxide of a metal;
- at least one carboxylate of a metal, said carboxylate comprising at least 10 carbon atoms.

[0035] Advantageously, the process of the invention comprises mixing by dry blending and/or melt compounding polymer (P), the oxide and the carboxylate.

[0036] Preferably, polymer (P), the oxide and the carboxylate are mixed by melt compounding.

[0037] Advantageously, polymer (P), the oxide and the carboxylate are melt compounded in continuous or batch devices. Such devices are well-known to those skilled in the art.

[0038] Examples of suitable continuous devices to melt compound the sulfone polymer composition of the invention are notably screw extruders. Thus, polymer (P), the oxide and the carboxylate and optionally other ingredients, are advantageously fed in powder or granular form in an extruder and the composition is extruded into strands and the strands are chopped into pellets.

[0039] Optionally, fillers, lubricating agents, heat stabilizer, anti-static agents, extenders, reinforcing agents, organic and/or inorganic pigments like TiO_2, carbon black, flame-retardants, smoke-suppressing agents may be added to the composition during the compounding step.

[0040] Preferably, polymer (P), the oxide and the carboxylate are melt compounded in a twin-screw extruder.

[0041] The composition can be further processed following standard methods for injection molding, extrusion, thermoforming, machining, and blow molding. Solution-based processing for coatings and membranes is also possible. Finished articles comprising the composition as above described can undergo standard post-fabrication operations such as ultrasonic welding, adhesive bonding, and laser marking as well as heat staking, threading, and machining.

[0042] Another object of the invention is an article comprising the polymer composition as above described.

[0043] Advantageously, the article is an injection molded article, an extrusion molded article, a shaped article, a coated article or a casted article. Preferably it is an injection molded article.

[0044] The articles according to the invention can be fabricated by processing the composition as above described following standard methods.

[0045] The present invention is described in greater detail below by referring to the Examples; however, the present invention is not limited to these examples.

Examples 1 to 5 :

Raw material:

[0046] RADEL® R 5600 polyphenylsulfone commercially available from Solvay Advanced Polymers, L.L.C., is a polyphenylsulfone obtained from the polycondensation of a 4,4'-dihalodiphenylsulfone and 4,4'-dihydroxydiphenyl.

Compounding :

[0047] Zinc Oxide, Zn stearate and Mg stearate were mixed with the RADEL® R 5600 polyphenylsulfone pellets and melt compounded using a 25 mm diameter twin screw double vented Berstorff extruder having an L/D ratio of 33/1. Details of quantities used in the examples are reported in Table 1.

Ionics extraction and analytical determinations

[0048] A sample of the compounded pellets from each of the formulations, as well as of RADEL® R 5600 polyphenylsulfone alone, have been submitted to ionics extraction according to the following procedure :

1.0 g of sample was placed in 5.0 ml of 18 mΩ water (Milli-Q available from Alltech Associates, Inc.). The samples were then placed in an oven at 85°C for 24 hours to extract the ions.

[0049] After 24 hours, the pellets have been separated and the water phase has been analysed by IC (Ion Chromatograpy), using the following analytical system :

Dionex DX500 Chromatography System;
Dionex GP50 Standard bore Gradient Pump;
Dionex ASRS Ultra 4 mm Suppressor;
Dionex CD20 Conductivity Detector;
Dionex AS11A Column, 4 mm;
Dionex AG11A Guard Column, 4 mm;
Dionex AS40 Autosampler, Inert Peek Flow Path.

[0050] The eluent used was 18 mΩ water, 0.2-0.4 mM KOH by provided by the EG40 Eluent Generator.
[0051] The ion chromatography was quantified using a minimum of 5-points linear calibration curve for each ion. Results are joined in Table 1.

Table 1

Component/ Property	Comparative example 1	Comparative example 2	Comparative example 3	Comparative example 4	Example 5
	No additive package	Additives package			
ZnO (% wt)		0	0	0.50	0.03
Zn stearate (%wt)		0	0.30	0	0.30
Mg stearate (% wt)		0.30	0	0	0
Composition aspect					
Transparency	Transparent	Opaque	Transparent	Transparent	Transparent
Ionics Extraction results (ng/g)					
Sulfate	730	56	180	68	62
Zinc	55	< 40	37	3400	80
Mg	n.d.	76	n.d.	n.d.	n.d.

Examples 6 to 8 :

Raw material :

[0052] UDEL® P3703 polysulfone commercially available from Solvay Advanced Polymers, L.L.C., is a polysulfone obtained from the polycondensation of a 4,4'-dihalodiphenylsulfone and bisphenol A.

Compounding :

[0053] Zinc Oxide and Zinc stearate were mixed with the UDEL® P3703 polysulfone pellets and melt compounded using a 25 mm diameter twin screw double vented Berstorff extruder having an L/D ratio of 33/1. Details of quantities used in the examples are reported in Table 2.

Ionics extraction and analytical determinations

[0054] Same procedure as followed for examples 1 to 5 was repeated for the compositions of examples 7 and 8, as well as for UDEL® P3703 polysulfone alone (comparative example 6). Results are joined in Table 2.

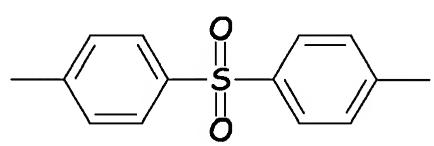
Table 2

Component/Property	Comparative example 6	Example 7	Example 8
Additives package			
ZnO (% wt)	No additive package	0.05	0.10
Zn stearate (% wt)		0.25	0.20
Ionics Extraction results (ng/g)			
Sulfate	3.0	0.29	0.30
Zinc	<5.0	<5.0	<5.0

Claims

1. Aromatic high glass transition temperature sulfone polymer composition comprising :

   - at least 90 % by weight, based on the total weight of the composition, of at least one aromatic sulfone polymer (P), wherein at least 50 % wt of the recurring units of the aromatic sulfone polymer (P) comprise at least one group of formula 1 :



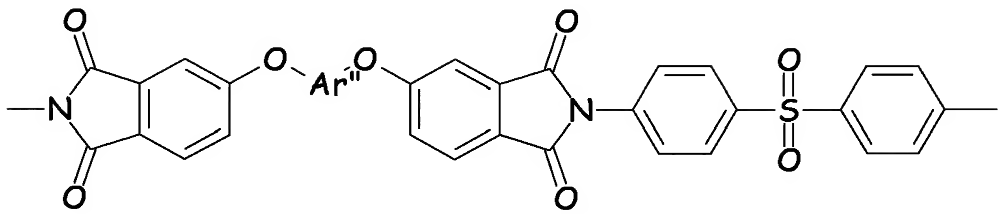
(formula 1);

   - at least one oxide of a metal;
   - at least one carboxylate of a metal, said carboxylate comprising at least 10 carbon atoms.

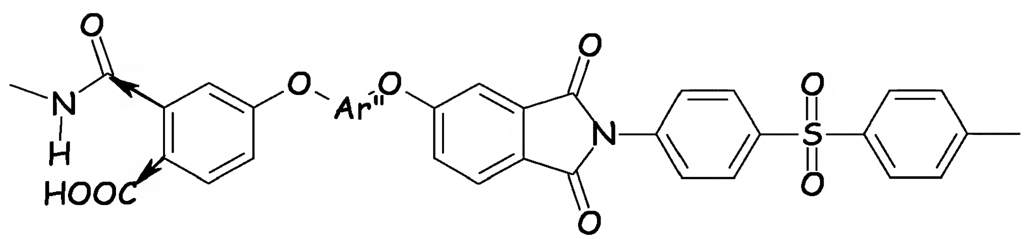
2. Aromatic high glass transition temperature polymer composition of Claim 1, wherein the oxide of a metal is an oxide of an alkaline-earth metal or of a group 12 transition metal.

3. Aromatic high glass transition temperature polymer composition of Claim 1 or 2, wherein the carboxylate of a metal is a carboxylate of an alkaline-earth metal or of a group 12 transition metal.

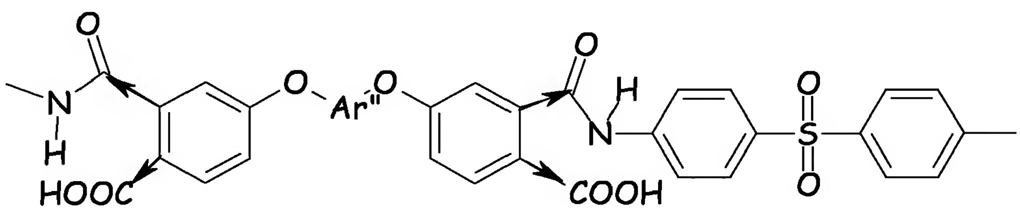
4. Aromatic high glass transition temperature sulfone polymer composition of anyone of preceding claims, **characterized in that** at least 50 % wt of the recurring units of the aromatic sulfone polymer (P) are recurring units (R1), in their imide form (R1-A) and/or amic acid forms [(R1-B) and (R1-C)] :



(R1-A)



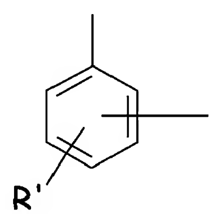
(R1-B)



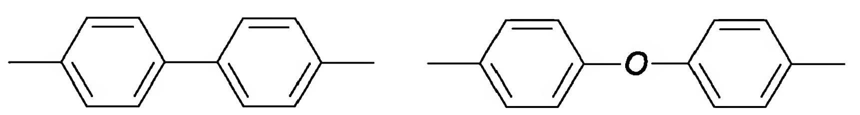
(R1-C)

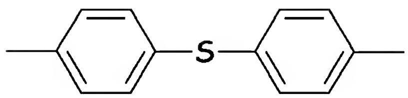
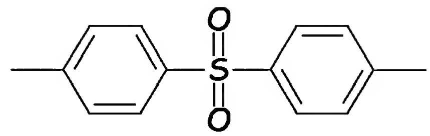
wherein :

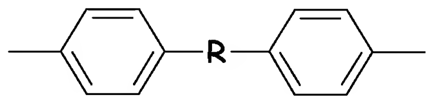
- the → denotes isomerism so that in any recurring unit the groups to which the arrows point may exist as shown or in an interchanged position;
- Ar" is chosen among the following structures :



with the linking groups being in ortho, meta or para position and R' being a hydrogen atom or an alkyl radical comprising from 1 to 6 carbon atoms ,

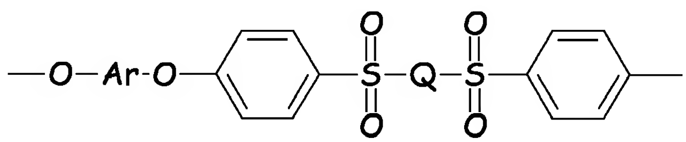


 ,  ,

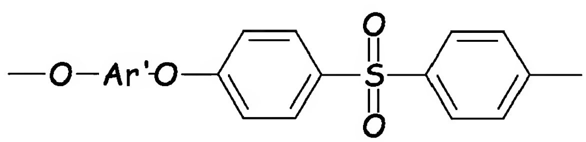


with R being an aliphatic divalent group of up to 6 carbon atoms, such as methylene, ethylene, isopropylene, and mixtures thereof.

5. Aromatic high glass transition temperature sulfone polymer composition according to anyone of claim 1 to 3, **characterized in that** at least 50 % wt of the recurring units of the aromatic sulfone polymer (P) are recurring units (R2) and/or recurring units (R3) :



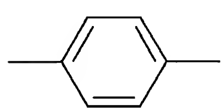
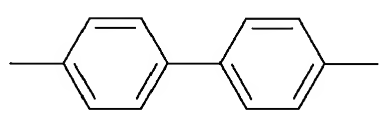
(R2)

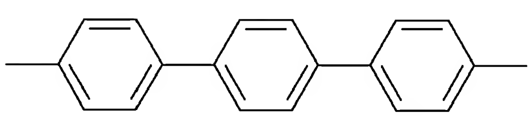
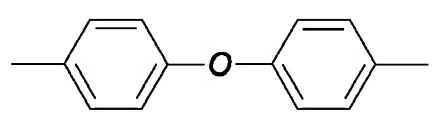


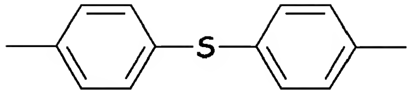
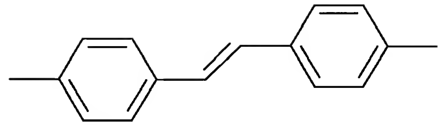
(R3)

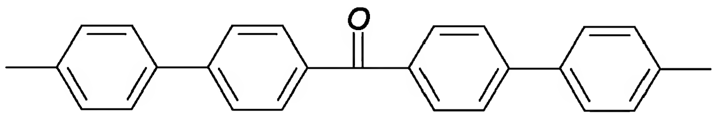
wherein :

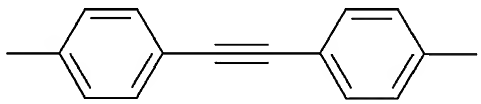
- Q is a group chose among the following structures :

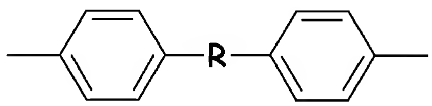
 ,  ,

 ,  ,

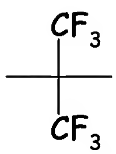
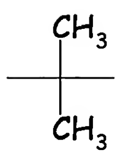
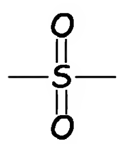
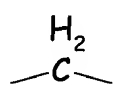
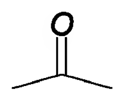
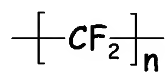
 ,  ,

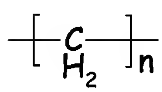
 ,

 ,



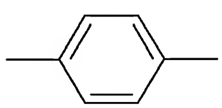
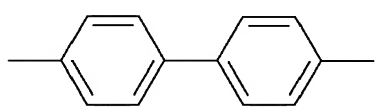
with **R** being :

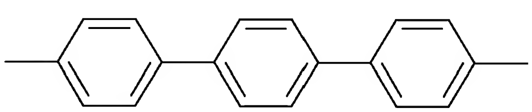
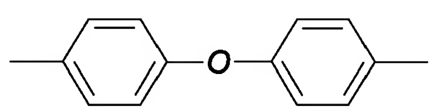
 ,   ,  ,  ,  ,

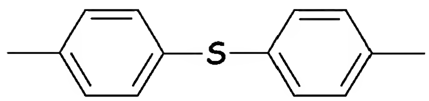
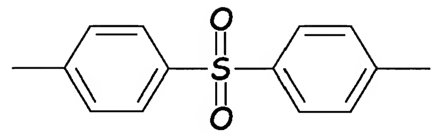


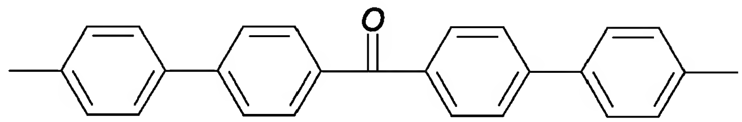
with n = integer from 1 to 6, or an aliphatic divalent group, linear or branched, of up to 6 carbon atoms; and mixtures thereof;

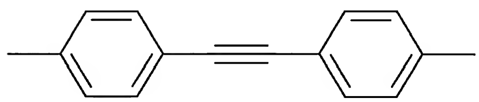
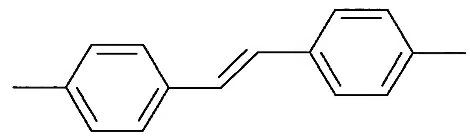
- **Ar** is a group chosen among the following structures :

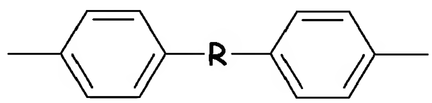
 ,  ,

 ,  ,

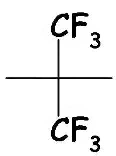
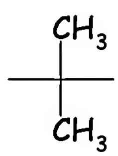
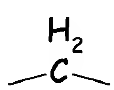
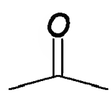
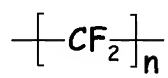
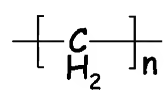
,
,

,

,
,

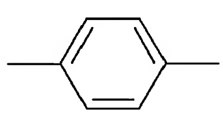
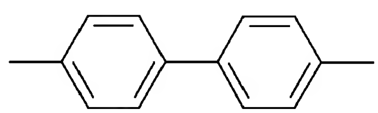


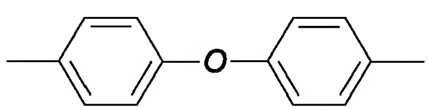
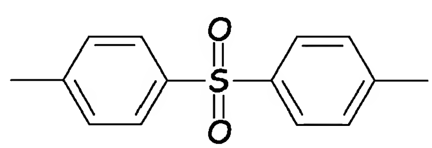
with **R** being :

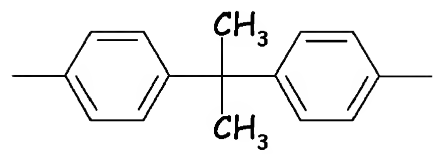
,

,
,
,


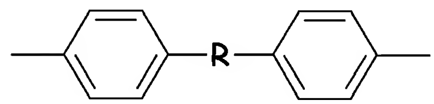
with n = integer from 1 to 6, or an aliphatic divalent group, linear or branched, of up to 6 carbon atoms; and mixtures thereof;

- **Ar'** is a group chosen among the following structures :

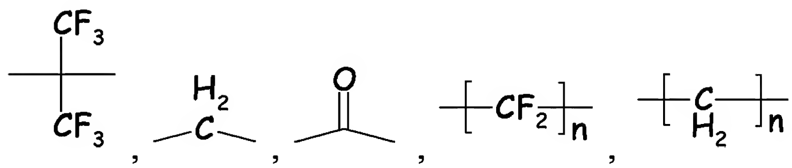
,
,

,
,





with **R** being :



with n = integer from 1 to 6, or an aliphatic divalent group, linear or branched, of up to 6 carbon atoms; and mixtures thereof.

6. Aromatic high glass transition temperature sulfone polymer composition according to anyone of preceding claims, **characterized in that** it comprises from 0.02 to 0.10 % wt, based on the total weight of the composition, of the oxide.

7. Aromatic high glass transition temperature sulfone polymer composition according to anyone of preceding claims, **characterized in that** it comprises from 0.05 to 0.35 % wt, based on the total weight of the composition, of the carboxylate.

8. Aromatic high glass transition temperature sulfone polymer composition according to anyone of preceding claims, **characterized in that** the weight ratio between the oxide and the carboxylate is from 0.1 to 0.5 wt/wt.

9. Process for manufacturing the high glass transition temperature sulfone polymer composition according to anyone of preceding claims, which comprises mixing :

- at least one aromatic sulfone polymer (P);
- at least one oxide of a metal;
- at least one carboxylate of a metal, said carboxylate comprising at least 10 carbon atoms.

10. Article comprising the aromatic high glass transition temperature sulfone polymer composition of anyone of Claims 1 to 8.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 05 10 1408

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
X	DATABASE CAPLUS [Online] CHEMICAL ABSTRACTS SERVICE, COLUMBUS, OHIO, US; 29 November 1995 (1995-11-29), AKYAMA, MASAMI: "Heat-resistant and electrically conductive resin compositions and heat-resistant integrated circuit (IC) trays with good dimensional stability using them" XP002337661 retrieved from STN Database accession no. 1995:947329 * abstract * & JP 07 238216 A (PLAS TECH CORP) 12 September 1995 (1995-09-12)	1-10	C08K3/22 C08K5/09 C08K13/02
X	US 4 420 528 A (OKIYAMA ET AL) 13 December 1983 (1983-12-13) * claims *	1-10	
A	EP 0 727 456 A (SUMITOMO CHEMICAL COMPANY, LIMITED; SUMITOMO CHEMICAL COMPANY LIMITED) 21 August 1996 (1996-08-21) * claims *	1-10	

TECHNICAL FIELDS SEARCHED (Int.Cl.7)

C08K

The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	25 July 2005	Andriollo, G

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

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E : earlier patent document, but published on, or after the filing date
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& : member of the same patent family, corresponding document

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 05 10 1408

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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25-07-2005

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP 7238216 A	12-09-1995	NONE	
US 4420528 A	13-12-1983	JP 1652707 C	30-03-1992
		JP 3003216 B	18-01-1991
		JP 57014849 A	26-01-1982
EP 0727456 A	21-08-1996	JP 8217976 A	27-08-1996
		DE 69603097 D1	12-08-1999
		DE 69603097 T2	16-12-1999
		EP 0727456 A1	21-08-1996
		US 6124390 A	26-09-2000

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

PUB-NO: EP001695994A1

DOCUMENT-IDENTIFIER: EP 1695994 A1

TITLE: Aromatic high glass temperature sulfone polymer composition

PUBN-DATE: August 30, 2006

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REICHMANN, MARK G	US

ASSIGNEE-INFORMATION:

NAME	COUNTRY
SOLVAY ADVANCED POLYMERS L L C	US

APPL-NO: EP05101408

APPL-DATE: February 24, 2005

PRIORITY-DATA: EP05101408A (February 24, 2005)

ABSTRACT:

The invention relates to an aromatic high glass transition temperature polymer composition comprising : at least one aromatic sulfone polymer (P); at least one oxide of a metal; at least one carboxylate of a metal, said carboxylate comprising at least 10 carbon atoms. Preferably, the aromatic sulfone polymer (P) is chosen among

the group consisting of polybiphenyldisulfone, polysulfone, polyphenylsulfone, polyethersulfone, copolymers and mixtures thereof.